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Docket No. 24180-096013 PATENT

IN THE UNITED STATES PATENT AND TRADEMARK OFFICE

In re Eckstein, et al., U.S. Patent Application No. 08/886,881

For: **Improved Structures of Polymers Made From Single Site Catalysts**

Filed: July 2, 1997

Group Art Unit: 1773

Examiner: D. Tarazano

Rule 131(b) Declaration of Gregory K. Jones

Commissioner of Patents and Trademarks
Washington, D.C. 20231

Dear Sir:

This declaration is being submitted under 37 CFR §1.131 to show prior invention over U.S. patent No. 5,376,439 to Hodgson et al. The undersigned co-inventor declares the following:

1. I am one of the named co-inventors for U.S. Patent Application No. 08/886,881, which was filed on July 2, 1997 as a continuation of U.S. Patent Application No. 08/653,520, filed May 15, 1995, now abandoned, which is a continuation of U.S. Patent Application No. 08/082,226, filed June 24, 1993, now abandoned.

2. Claims 1, 3, 7, 8, 10-16, 18, 35, 37, 41-49, 51, and 98-106 are pending in U.S. Patent Application No. 08/886,881. Independent claim 1 relates to a film structure comprising at least two layers wherein a first layer comprises a barrier material and further wherein a second layer comprises a polymer formed by the polymerization reaction with a single site catalyst. Further, independent claim 35 relates to a film structure comprising at least two layers wherein a first layer comprises a barrier material and further wherein a second layer comprises a polymer formed by the polymerization reaction with a metallocene catalyst system.

3. The primary reference, U.S. Patent No. 5,376,439 to Hodgson et al. issued on December 27, 1994 from an application filed on March 29, 1994, which was a continuation of U.S. Patent Application No. 945,769, which was filed on September 16, 1992, now abandoned.

4. Film structures as defined in the claims of the present invention had been conceived prior to September 16, 1992, the effective date of Hodgson et al. Specifically, Paragraphs 6-12 and Exhibits A-G as described below show conception of the claimed invention prior to September 16, 1992, the effective date of the reference, coupled with due diligence from prior to the reference date to a subsequent constructive reduction to practice (i.e., the filing date of U.S. Patent Application No. 08/082,226, dated June 24, 1993).

5. The attached copies are true copies of original Experiment and Development Orders and notebook entries. However, the experimental order numbers ("E-numbers") of some of the copies are blacked out because the E-numbers had been highlighted and photocopied. The true E-numbers of the documents are presented in the following paragraphs.

6. Exhibit A, dated August 18, 1992, shows Experiment and Development Order No. E-15476-92 ("Order No. E-15476-92") relating to five layer film structures, each comprising a first layer comprising a barrier material (Saran blend 3649-00) and a second layer comprising a polymer formed by the polymerization reaction with a single site catalyst or a metallocene catalyst system (Exxon SLP-0179 or Exxon SLP-9012). Order No. E-15476-92 was prepared and requested by J. Zheng, a named co-inventor of the subject matter of present invention, on August 18, 1992 and was further approved by Roger L. Kaas on August 19, 1992. As stated, the objective of Order No. E-15476-92 was to use "Exxon VLDPE to replace Dow ULDPE in five layer structure [sic]." The Exxon resins utilized in the above-identified Experiment and Development Order (SLP-0179 and SLP-9012) are polyethylene copolymers made using a single site catalyst or metallocene catalyst system, known by Exxon as Exxon Exxpol technology.

7. Exhibit B, dated August 18, 1992, shows a notebook page titled "Five Layer Shrink Bag Modifications - VLDPE." The stated objectives are: "1) determine the five layer shrink bags' performance using VLDPE replacing ULDPE; 2) eventually cost reduction (down gauging); and 3) improved sealability and toughness." The notebook entry was prepared by and initialed by J. Zheng. Further, the notebook entry indicates that a trial run took place on 9-17-92 and was successful. Further, Exhibit B shows a handwritten copy of Order No. E-15476-92

pasted to the notebook page that eventually became Order No. E-15476-92, as shown in Exhibit A.

8. Exhibit C shows a web page dated October 3, 2000 called "How EXXPOL Metallocene Catalysts Work." The purpose of this web page is to show that the Exxon Exxpol technology shown in Order No. E-15476-92 is, in fact, single-site catalyzed or metallocene catalyzed technology and further to show comparisons of the Exxon Exxpol technology against prior art technologies, such as Ziegler-Natta technology.

9. Exhibit D, dated August 19, 1992, shows Experiment and Development Order No. E-15474-92 ("Order No. E-15474-92") related to EB (electron beam) cross-linking of VLDPE, having the stated objective: "To determine the EB cross-linking of butene and hexene based Exxon VLDPE." The materials used were SLP-0179 and SLP-9012, which are both single site catalyzed polymer materials. Order No. E-15474-92 was prepared by J. Zheng and approved by Roger L. Kaas on September 19, 1992.

10. Exhibit E, dated December 11, 1992, shows Experiment and Development Order No. E-15511-92 ("Order No. E-15511-92"), titled "Exxon Exact Polymer Evaluation - Shrink Bag Application". The objective was "to determine the processibility of the EB cross-linking and the performance properties of Exact polymers in three layer shrink bag application [sic]." Variables 3-4 and 6-7 have the Exxon Exact single-site catalyzed materials in layers of three layer structures, each also having a layer comprising a barrier material (Saran blend). Order No. E-15511-92 was prepared and requested by J. Zheng and approved by Roger L. Kaas on December 11, 1992.

11. Exhibit F, dated February 8, 1993, shows Experiment and Development Order No. E-15408-93 ("Order No. E-15408-93"), titled "Dow CGCT Polymer Evaluation - Shrink Bag Application." The objective was "to determine the processibility, the EB cross-linking and the performance properties of Dow CGCT/Constrained Geometry Catalyst Technology) polymers in three layer shrink bag application [sic]." Variables 3 and 4 of Order No. E-15408-93 are three layer structures having a layer of a barrier material (Saran Blend 3649) and a layer comprising a polymer formed by the polymerization reaction of a single site catalyst (Dow XUR-1567-48562A37 and Dow XUR-1564-48562A23). Order No. E-15408-93 was prepared and requested by J. Zheng on February 8, 1993 and approved by Roger L. Kaas on February 12, 1993. In

addition, Dow CGCT polymers are polyethylene copolymers made by a single site catalyst or metallocene catalyst system.

12. Exhibit G, dated March 2, 1993, shows Experiment and Development Order No. E-15413-93, titled "3-Layer Saran Sealability and Toughness" ("Order No. E-15413-93"). The objective was to "evaluate Dow Insite Polymer and modify the current 80/10/10 formulation to improve the sealability and toughness of 3-layer layer shrink bags." Variables 2-6 show three layer film structures having a layer of barrier material (Saran Blend 3653) and a layer comprising a polymer formed by the polymerization reaction with a single site catalyst (Dow XUR-1567-58462A37). Order No. E-15413-93 was prepared by J. Zheng, requested by K. Lind, a named co-inventor of the subject matter of the present invention, and approved by Roger Kaas on March 2, 1993. Dow Insite polymers are polyethylene copolymers made by a single site catalyst or metallocene catalyst system.

CONCLUSION

13. Exhibits A-G show a conception of the present invention prior to the effective date of U.S. Patent No. 5,376,439 coupled with due diligence from prior to the reference date to a subsequent reduction to practice. Therefore, Exhibits A-G establish prior invention of the claimed subject matter prior to September 16, 1992, the effective priority date of U.S. Patent No. 5,376,439. However, this declaration is not to be construed as an assertion that an earlier date of conception or reduction to practice does not exist.

14. The declarant further declares that all statements made herein of his own knowledge are true and that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both under Section 1001 of Title 18 of the United States Code, and that such willful false statements may jeopardize the validity of the application of any patent issuing thereon.

Respectfully Submitted,

Date: December 4, 2001

Gregory K. Jones

CHI99 3756011-1.024180.0096

EXPERIMENT AND DEVELOPMENT ORDER

Prepared By:	J. Zheng	Experimental Number:	[redacted]
Requested By:	J. Zheng	Charge Cost To:	490102-91611
Date:	8/18/92	Pr ject No.:	MP-800
Industry:	Meat	Machines Involved In Cost:	
Customer:			
Shipping:			

Title: Five Layer Shrink Bag Modification - VLDPE

Objective: Using Exxon VLDPE to replace Dow ULDPE in five layer structure.

Instructions:

Materials:
Saran blend 3649-00
ULDPE: Dow 4201 RMS 11188
EVA-ULDPE Blend: 3652-00
VLDPE: Exxon SLP-0179
Exxon SLP-9012
EVA: Exxon 97.06 RMS 11459

Five Layer Structure:

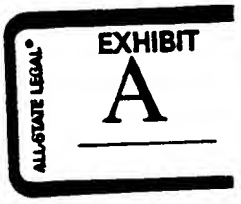
	30 ga.	30 ga.	20 ga.	90 ga.	30 ga.
Variable 1 (control):	3652-00	/ 4201 /	3649-00	/ 11459 /	4201
Variable 2:	3652-00	/ 4201 /	3649-00	/ 11459 /	SLP-0179
Variable 3:	3652-00	/ 4201 /	3649-00	/ 11459 /	SLP-9012
Variable 4:	3652-00	/ SLP-0179 /	3649-00	/ 11459 /	SLP-0179
Variable 5:	3652-00	/ SLP-9012 /	3649-00	/ 11459 /	SLP-9012

PP - Extrusion: Produce 1,000 ft. of each variable (14" wide)

EB:
- Prepare 3 composite rolls
- EB treat two passes at total dose 4, 5.3 and 6 Mrad for three rolls

Estimated Cost: $500.00 Approval: Roger L. Kaas Date: 8/19/92
Assigned To: J. Zheng
Assisted By: D. Cowling

Distribution: R. Germonprez, B. Steen, Library, L. Christenson, J. Eckstein, K. Peterson, R. Kaas, J. Zheng (2), D. Cowling (2)



8-18-92

Title: Five layer Shrink bag modifiction - VLDPE

Objective: ① determine the 5-layer shrink bags performance using VLDPE replacing ULDPE.

② Eventually cost reduction. [downgaging]

③ Improve sealability and toughness

EXPERIMENT AND DEVELOPMENT ORDER

E-15V76-92

Title Five layer Shrink bag Modification — VLDPE

OBJECTIVE: ... using Exxon VLDPE to replace Dow ULDPE in five layer structure.

TO BE FILLED OUT ON COMPLETION OF JOB

EXPERIMENT COMPLETION DATA

II. ... Extrusion

Produce ... of each variable

IV. EB

Prepare 3 composite rolls

· EB ... for ... rolls

INSTRUCTIONS:

I. Materials

Surlyn Dow ... 1649-00

ULDPE Dow ...

EVA-ULDPE Blend ...

VLDPE: Exxon 3LP-0179

Exxon 3LP-9006

EVA: Exxon ...

II. Five layer Structure

Variable 1 (control): /[illegible]/[illegible]/[illegible]/[illegible]/[illegible]/

Variable 2. /[illegible]/[illegible]/[illegible]/[illegible]/3LP-0179/

/[illegible]/[illegible]/[illegible]/[illegible]/3LP-9006/

3. /[illegible]/[illegible]/[illegible]/[illegible]/3LP-0179/

4. /[illegible]/[illegible]/[illegible]/[illegible]/3LP-9006/

5.

STOCK AND PRODUCTION DISPOSITION DATA

* E - Trial written at PPCO 9-17-92. The trial was successful

EXHIBIT B

ALL-STATE LEGAL

ExxonMobil Chemical

ExxonMobil Chemical Categories

EXXPOL™ | Home | New Chemical Catalysts | How They Work | Key Features | Product Properties | Products Made with EXXPOL

Licensing Technolo

- Technology Ov
- Polymers Techn Licensing
- Exxpol Techno
- Aromatics and Olefins Licens

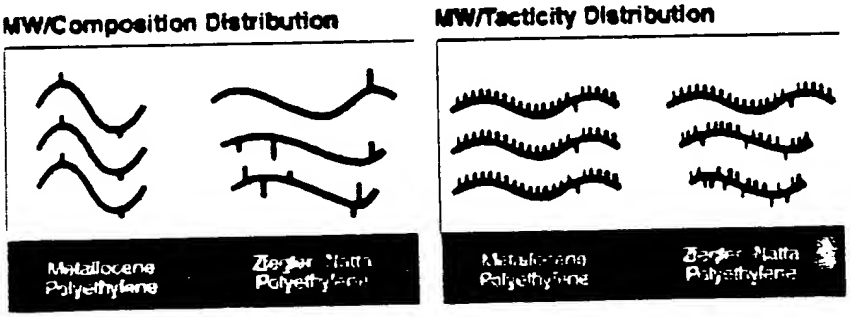
How EXXPOL Metallocene Catalysts Work

Catalysts can be compared to tiny hands or "templates" that initiate and guide the assembly of a polymer.

The template "grabs" individual olefin molecules and links them together to form a polymer chain.

Prior to metallocenes, catalysts were chemical blends made up of slightly different molecules that each assembled the polymer chain in different ways. With this variety of templates, it was difficult to control the structure of the resulting polymer molecules, causing those molecules to have slightly different and inconsistent properties.

EXXPOL metallocene catalysts each consist of identical molecules, or templates, which behave in the same way, so each polymer chain produced is uniform, resulting in polymer consistency.



EXXPOL metallocene catalysts offer "single-site" catalysis:

- a single type of catalytic site rather than multiple types of sites
- producing a single type of polymer chain.

What's more, scientists and engineers can change the structure of the catalyst molecule and the environment around its catalytic site to build different polymeric structures with specific properties, such as increased toughness or better clarity.

With the ability to produce extremely uniform polymers and the flexibility to design those polymers to provide the desired attributes, metallocene catalysts offer new opportunities to create polyolefins that precisely meet market needs.

Form No. 3096
Rev. 8-53

EXPERIMENT AND DEVELOPMENT ORDER

Prepared By J. Zheng
Requested By
Date 8-19-92
Industry
Customer:
Shipping Instructions:

Experimental Number E-15474-92
Sales Order No.
Charge Cost To 490102-9611
Dept., Problem, or Devel. No. LR-200
Machines Involved in Cost:
PP - Rheology Extruder
ESI - EB.

Title EB Crosslinking of VLDPE

OBJECTIVE: Indicate purpose of experiment, title or description of product desired, and end use requirements.

To determine the EB crosslinking of future and Exxon based Exxon VLDPE.

INSTRUCTIONS: List materials to be used, Plants involved with detailed manufacturing instructions and disposition of finished goods and excess raw materials.

Materials:

Exxon SLP-0.179 25 lb
" SLP-9012 25 lb
[redacted]
Dow Attane 4201 25 lb Cost: $75.00

EB (ESI 250 Kv Unit) $250.00/hr Cost: $375.00
Total Cost: $450.00

PP- Rheology Extruder
Produce [redacted] 30 ft. 7 to 8 mil thick sheet of each following variables.

			density
Variable	1	SLP-0.179	0.910
	2.	SLP-9012	0.911
	3	Attane 4201	0.912

Foreman's Instructions: All Labor and Materials Used on This Experiment must be Reported on Your Labor, Production and Mate... Reports under the Above Order No.

Estimated Cost: $450.00
Assigned To:

Approval [signature] Date 4-15-93

EXHIBIT D

ALL-STATE LEGAL

EXPERIMENT AND DEVELOPMENT ORDER

Prepared By: J. Zheng
Requested By: J. Zheng
Date: 12/11/92
Industry:
Customer:
Shipping:

Experimental Number: [redacted]
Charg Cost To: 490102-91611
Project No.: LR-200
Machines Involved In Cost:
PP - Saran
ESI or PPDI - EB

Title: Exxon Exact Polymer Evaluation - Shrink Bag Application

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Objective: To determine the processability, the EB crosslinking and the performance properties of exact polymers in three layer shrink bag application.

Instructions:

Materials
Saran blend
EVA/ULDPE blend
Dow Attane 4201
Exxon Exact 3025 (T_m = 103°C)
Exxon Exact 3028 (T_m = 92°C)

Total Cost = $500.00

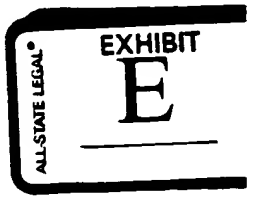
PP - Saran:
Produce three layer structure films, 2.3 mil thick and 14-1/2" wide with following variables.

Variable 1: (control) EVA/UL//Saran//EVA/UL-3651

Variable 2: EVA/UL//Saran//4201

Estimated Cost: $500.00 Approval: Roger L. Kaas Date: 12/11/92
Assigned To: J. Zheng
Assisted By: J. Holcomb

Distribution:
R. Germonprez
B. Steen
[redacted]
L. Christenson
K. Peterson
J. Eckstein
R. Kaas
J. Britt FPFF 06H
S. Godsil (7) PPDI
J. Holcomb (2)
J. Zheng (2)



-cont-

Variable 3: EVA/UL//Saran//3025

Variable 4: EVA/UL//Saran//3028

Variable 5: 4201/Saran//4201

Variable 6: 3025//Saran//3025

Variable 7: 3028//Saran//3028

* DR = 3.5 BUP = 3.8

EB - ESI or PPDI
TBD

EXPERIMENT AND DEVELOPMENT ORDER

Prepared By: J. Zheng
Requested By: J. Zheng
Date: 2/8/93
Industry:
Customer:
Shipping:

Experimental Number: [redacted]
Charge Cost To: 0-042-1284 763-02
Project No.: LR-200
Machines Involved In Cost:
PP - Saran
PPDI - EB

Title: Dow CGCT Polymer Evaluation - Shrink Bag Application

Objective: To determine the processability, the EB crosslinking and the performance properties of Dow CGCT/Constrained Geometry Catalyst Technology) polymers in 3-layer shrink bag application.

Instructions:

Materials
Saran Blend 3649
EVA/ULDPE Blend 3651
Dow Attane 4201
Dow XUR-1567-48562A37 (MI = 1.0 Density = 0.908)
Dow XUR-1564-48562A23 (MI = 1.0 Density = 0.895)

PP - Saran
Produce 3-layer structure films, 2.3 mil thick and 14-1/2" wid with following variables.

Variable 1: (control)
EVA/ULDPE // Saran // EVA/ULDPE

Variable 2:
EVA/ULDPE // Saran // 4201

Variable 3:
EVA/ULDPE // Saran // XUR-1567-48562A37

Variable 4:
EVA/ULDPE // Saran // XUR-1564-48562A23

PPDI - EB

Estimated Cost: $500.00 Approval: Roger L. Kaas Date: 2/12/93
Assigned To: J. Zheng
Assisted By: J. Holcomb

Distribution:
R. Germonprez
B. Steen
Library
L. Christ ns n
K. Peterson
R. Kaas
J. Britt FPFF 06H
J. Zheng (2)
S. Godsil (7) PPDI
J. H lcomb (2)

PO 2/17/93

EXPERIMENT AND DEVELOPMENT ORDER

Prepared By: J. Zh ng
Requested By: K. Lind
Date: 3/2/93
Industry: Meat
Customer: Not to Ship
Shipping: TBA

Experimental Number [redacted]
Charg Cost To: 0-042-1284 763-02
Pr ject No.: LR-200
Machines Inv lved In Cost:
PPDI - Saran Extrusion 3-layer
- EB
- Bag Machine

Title: 3-layer Saran Sealability and Toughness

Objective: The purpose of this E-number is to evaluate Dow Insite Polymer and modify the current 80/10/10 formulation to improve the sealability and toughness of 3-layer shrink bags.

Instructions:

Materials
Saran Blend: 3653-00
EVA, ULDPE Blend: 3652-00
Exxon LD 712.06 (10% VA, 0.3 MI)
Exxon LD 318-92 (9.0% VA, 2.2 MI)
Dow XUR-1567-58462A37 (1.0 MI, density = 0.908)

Blending
Blend 300 lbs. of inner Dow PE, EVA blends for Variables 3, 4, 5 and 6 at PTNW and ship to PPDI.

PPDI - Saran (3-layer Dial)
Produce two 4M ft. roll of each of the following structures at 13" width at 230 ga. per Spec. Z-2655.

Estimated Cost: $1,500.00 Approval: Roger L. Kaas Date: 3/2/93
Assigned To: J. Zheng
Assisted By:

Distribution:
R. Germonprez
B. Steen
Library
L. Christenson
K. Peterson
S. Godsil (7) PPDI
R. Kaas
J. Britt YPFF 06H
K. Lind (2)
[redacted] (2)
J. Eckstein

EXHIBIT G

PPDI - Saran -cont.-

Variable	Outer	Core	Inner
1	3652	3653	3652
2	3652	3653	Dow XUR-A37
3	3652	3653	90/10 A37/712.06
4	3652	3653	80/20 A37/712.06
5	3652	3653	50/50 A37/712.06
6	3652	3653	80/10/10 712.06/A37/318.92

Total Gauge 230 100-130 ga. 20 ga. 80-110 ga.

PPDI - EB Line
1. EB 1 roll each variable to 4.0 Mrad per Spec. M-803.
2. EB 1 roll each variable to 5.3 Mrad per Spec. M-811.

PPDI - Bag Machine
Produce 500 13 X 26" bags each variable per Spec. B-1233
(12 total cartons)

Testing
PPDI per Spec. Z-2655, M-808, M-811 and B-1233
PTNW per J. Zheng